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(54) **Nitrogen oxide traps**

Stickstoffoxid-Abscheider

Séparateurs pour oxyde d'azote

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Description

[0001] This invention relates to nitrogen oxide traps employed in the exhaust system of an internal combustion engines to absorb nitrogen oxides during lean-burn operation.

[0002] Catalysts are employed in the exhaust systems of automotive vehicles to convert carbon monoxide, hydrocarbons, and nitrogen oxides (NO_x) produced during engine operation into more desirable gases. When the engine is operated in a stoichiometric or slightly rich air/fuel ratio, i.e., between about 14.7 and 14.4, catalysts containing palladium or platinum are able to efficiently convert all three gases simultaneously. That is, the carbon monoxide and hydrocarbons are oxidised to carbon dioxide and water and the NO_x is reduced to nitrogen. Hence, such catalysts are often called "three-way" catalysts. It is desirable, however, to operate the engine in a "lean-burn" condition where the A/F ratio is greater than 14.7, generally between 19 and 27, to realise a benefit in fuel economy. While such three-way catalyst containing palladium or platinum are able to convert carbon monoxide and hydrocarbons during lean-burn (excess oxygen) operation, they are not efficient in reducing the NO_x .

[0003] It has become known that certain materials like potassium or strontium (hereafter "alkaline materials") in combination with platinum are capable of storing (absorbing) nitrogen oxides under conditions of excess oxygen. The widely held mechanism for this phenomena is that the platinum first oxidises NO to NO_2 and the NO_2 subsequently forms a nitrate complex with the alkaline material. In a stoichiometric or rich environment, the nitrate is thermodynamically unstable, and the stored NO_x is released. NO_x then catalytically reacts with reducing species in the exhaust gas to form N_2 . These so-called " NO_x traps" are currently receiving considerable attention because they have application for removing NO_x from the exhaust gas of internal combustion engines during lean burn operation.

[0004] The conventional method of preparing these traps involves impregnating the platinum and alkaline material precursors together into a porous support material such as alumina. This washcoat material may then be applied to a honeycomb substrate through which the gases pass. However, we have found that the nitrogen oxide trap performance can be significantly improved by forming the washcoat in a different manner according to the present invention as described below.

[0005] European Patent Application 0716876 A1 is concerned with a catalyst for purifying exhaust gases, which is composed of a catalyst substrate, a first porous support layer supporting barium as a NO_x -occluding material and palladium, which is formed on the catalyst support, and a second porous support layer supporting platinum which is formed on the first porous support layer.

[0006] European Patent Application 0666103 A1 de-

scribes a catalyst for purifying exhaust gases including a porous support, an NO_x storage component including at least one member selected from the group consisting of alkaline-earth metals, rare-earth elements and alkali metals, and, loaded on the porous support, a noble metal catalyst ingredient adjacent to the NO_x storage component.

[0007] This invention is directed to a nitrogen oxide (NO_x) trap useful to trap nitrogen oxides during the lean-burn (excess-oxygen) operation of an internal combustion engine. This trap comprises distinct catalyst phases of: (a) a porous support loaded with 0.1 to 5 weight % catalyst comprising platinum; and (b) another porous support loaded with 2 to 30 weight % catalyst of at least one material selected from the group consisting of alkali metal elements and alkaline earth elements, the weight % of catalyst being based on the weight of the porous support. The trap comprises an intimate mixture of catalyst phases (a) and (b) rather than separate layers of catalyst phases (a) and (b). If more than one catalyst material (b) is selected, they may be provided together on one porous support or on individual porous supports.

[0008] In another aspect, the invention is an internal combustion engine exhaust gas catalyst system comprising the NO_x trap. The trap is arranged in the exhaust system and absorbs NO_x when the air/fuel ratio of exhaust gas flowing into said trap is lean and releases the absorbed NO_x when the oxygen concentration in the exhaust gas is lowered, as during stoichiometric operation of the engine. According to another aspect, the invention is a process for trapping nitrogen oxides using the nitrogen oxide trap of the present invention disclosed above.

[0009] Advantageously, we have found that NO_x traps according to the present invention have improved NO_x sorption efficiency as compared to conventional NO_x traps where materials like platinum and alkaline materials are loaded together on the same support. We suspect that a deleterious interaction takes place in conventional traps between the platinum and alkaline materials when they share the same support material and hence are in intimate contact with each other.

[0010] The invention will now be described, by way of example, with reference to the accompanying drawings, in which:

Figs. 1A (layered) and 1B (intimate mixture) are schematic drawings showing NO_x traps according to embodiments of the present invention;

Fig. 2 is a graph showing the nitrogen oxide trapping efficiency of NO_x trap A (comparative example) and NO_x trap B embodying the present invention; and Fig. 3 is a graph showing the nitrogen oxide trapping efficiency of NO_x trap B and NO_x trap C, both embodiments of the present invention.

[0011] This invention, according to one aspect, is directed to an internal combustion engine exhaust gas catalyst system comprising a nitrogen oxide trap. The

NO_x trap comprises catalysts of platinum and at least one material selected from alkali metal elements and alkaline earth metal elements. According to the present invention, it is critical that the catalyst comprising platinum is loaded on a different porous support than that carrying the alkali metal elements or alkaline earth metal elements.

[0012] In forming catalyst phase (a), a loading of about 0.1 to 5 weight percent platinum catalyst, preferably between about 0.5 and 2 percent platinum, is employed, based on the weight of the support on which it is loaded. In forming catalyst phase (b), a loading of about 2 to 30 weight percent catalyst selected from alkali metal and alkaline earth metals, preferably between about 10 and 20 percent such materials, is employed, based on the weight of the support on which these materials are loaded. Exemplary of alkali metal elements which may be employed include, but are not limited to, potassium, sodium, cesium, and lithium. Exemplary of alkaline earth elements which may be employed include, but are not limited to, strontium, calcium, and barium. In forming catalyst phase (b), if more than one such catalyst is employed, e.g., potassium and barium, they may be provided together on the same porous support or on their own individual porous supports. These distinct catalyst phases (a) and (b) may be provided as an intimate mixture thereof or as layers in the NO_x trap.

[0013] Each catalyst phase used in the nitrogen oxide trap comprises a catalyst loaded on a porous support (washcoat) material, which is a high surface area material like alumina, preferably being γ -alumina. Still other types of washcoat materials which may be used at the high operating temperatures associated with an internal combustion engine exhaust system include, but are not limited to, zeolite, zirconia, alpha alumina, cerium oxide (ceria), and magnesium oxide. Such washcoat materials, useful for carrying catalyst materials, are well known to those skilled in the art. The choice of the particular porous support (washcoat) material is not critical to this invention. Desirably, the support material has a surface area between about 10 and 300 m²/g. Each catalyst may be provided on the same or different type of porous support material. For example, according to one embodiment, the platinum catalyst may be loaded on ceria and the barium catalyst may be loaded on alumina. Preferably, however, the first porous support and the second porous support used for the respective catalysts is of the same type and is γ -alumina.

[0014] Generally, to provide a catalyst onto a porous support, a catalyst precursor compound is impregnated onto the support from a solution thereof by incipient wetness techniques. The solution can be water or organic solvent based. One embodiment invention trap may comprise platinum and strontium catalysts each deposited on different γ -alumina support portions. According to such techniques, to load platinum onto γ -alumina, the alumina may be impregnated with an aqueous solution of hexachloroplatinic acid. After impregnation, the materi-

al may be dried and calcined. If the porous support is other than ceria, e.g., γ -alumina, it is desirable to also incorporate via impregnation some rhodium and/or ceria into the platinum containing phase. The preferred rhodium concentration would be between 1/5 and 1/10 (by weight) of the platinum loading. The preferred ceria loading would range from 2 to 20 weight % of the porous support.

[0015] Strontium may be provided on a porous support in the same way from, e.g., strontium nitrate. Hence, another portion of γ -alumina could be impregnated with an aqueous solution of strontium nitrate and then dried. As discussed above, catalyst phase (b) comprises at least one material selected from alkali metal and alkaline earth metals. Thus, for example, if two such materials like potassium and cesium are employed, each may be provided on their own porous support (which may be of the same or different type) or provided together onto a single portion of porous support. It is only critical that neither of these materials be provided on the porous support carrying the platinum catalyst.

[0016] For useful application in an exhaust system, these catalyst phases will be carried on a substrate of a high temperature stable, electrically insulating material. Typical of such substrate materials are cordierite, mullite, etc. The substrate may be in any suitable configuration, often being employed as a monolithic honeycomb structure, spun fibres, corrugated foils or layered materials. Still other materials and configurations useful in this invention and suitable in an exhaust gas system will be apparent to those skilled in the art in view of the present disclosure.

[0017] The catalyst phase materials are generally applied as a slurry (washcoat) thereof onto the substrate. The catalyst phase materials may be washcoated as a mixture thereof or in sequential steps to form layers of the phases on the substrate, in a manner which would be readily apparent to those skilled in the art of catalyst manufacture. In either case, after applying the washcoat to the substrate, the materials are generally dried and calcined at elevated temperature. Figs. 1 schematically show an intimate mixture embodiment (Fig. 1B) and a layered embodiment (Fig. 1A) of the present invention. The intimate mixture embodiment is preferred since it provides more efficient NO_x removal. We believe that this advantage results because NO₂ can be absorbed by the alkaline material phase (b) immediately after it is formed over the platinum phase (a), hence driving the thermodynamically constrained NO + 1/2O₂ = NO₂ reaction in the favourable direction. With the platinum and the alkali metal phases separated into distinct washcoat layers, in contrast, a NO₂ molecule which is formed over the platinum phase needs to first diffuse through the platinum containing washcoat layer before it can be adsorbed by the alkali metal. Under this scenario, the NO to NO₂ reaction is more thermodynamically constrained and NO_x sorption efficiency is lower. While this theory has been put forth to explain this advantage, neither its

accuracy nor understanding is necessary for the practice of the present invention.

[0018] The internal combustion engine catalyst system of this invention may include, in addition to the nitrogen oxide trap, another catalyst device such as a three-way catalyst containing, for example, palladium, platinum and rhodium, or palladium and rhodium. The three-way catalyst device can be placed upstream of the NO_x trap, hence closer to the engine. In such an arrangement, the three-way catalyst being closely mounted to the engine would warm up quickly and provide for efficient engine cold start emission control. Once the engine is warmed up, the three-way catalyst will remove hydrocarbons, carbon monoxide, and nitrogen oxides from the exhaust during stoichiometric operation and hydrocarbons and carbon monoxide during lean operation. The NO_x trap would be positioned downstream of the three-way catalyst where the exhaust gas temperature enables maximum NO_x trap efficiency. During periods of lean engine operation when NO_x passes through the three-way catalyst, NO_x is stored on the trap. The NO_x trap may be periodically regenerated by short periods or intervals of slightly rich engine operation. With the NO_x trap positioned downstream of the three-way catalyst in a more remote location relative the engine, it is protected against very high exhaust gas temperatures which could damage the trap. It may also be considered desirable to optionally place a second three-way catalyst downstream of the NO_x trap in order to provide for efficient reduction of the NO_x which desorbs from the trap during regeneration.

[0019] If rhodium and ceria are incorporated into the platinum containing phase of the trap washcoat as disclosed above, this NO_x trap may desirably be used without a three-way catalyst. In this case, the platinum/rhodium/ceria containing phase of the NO_x trap provides the three-way catalyst function and hence NO_x control under stoichiometric engine operation.

Example 1

[0020] For comparison, a nitrogen oxide trap (Trap A) was prepared not according to the present invention. It was prepared as follows: γ -alumina powder (100m²/g) was impregnated via incipient wetness with an aqueous solution of strontium nitrate. The impregnated powder was then dried (120°C) and calcined (500°C). The resulting material was then impregnated via incipient wetness with an aqueous solution containing hexachloroplatinic acid. The material was then subsequently dried (120°C) and calcined (500°C). The strontium nitrate and hexachloroplatinic acid concentrations were adjusted so as to provide 10 wt. % strontium and 2 wt. % platinum on the finished material. The resulting Pt/Sr/ γ -alumina powder was ball milled and mixed with distilled water to produce a slurry. The slurry was applied to a cordierite monolith (400 cell/in²) to obtain a 25 wt. % loading of Pt/Sr/ γ -alumina on the monolith. The monolith was subse-

quently dried at 120°C and calcined in air at 500°C.

[0021] A nitrogen oxide trap (Trap B) according to an embodiment of the present invention was prepared. It involved first preparing distinct phases of Sr/ γ -alumina and Pt/ γ -alumina and then forming an intimate mixture of these two phases and applying it to a monolith as follows. γ -alumina loaded with strontium was prepared by taking γ -alumina powder (100m²/g) and impregnating it via incipient wetness with an aqueous solution of strontium nitrate. The impregnated powder was dried at 120°C and calcined at 500°C. The strontium nitrate concentration was adjusted in order to provide for 20 wt. % strontium on the finished material. The platinum was loaded on alumina by impregnating another amount of γ -alumina powder of the type above with an aqueous solution of hexachloroplatinic acid by incipient wetness. The solution concentration was fixed so as to yield 4 wt. % platinum on the alumina. Following impregnation, the material was dried and calcined as above for the strontium. Equal parts by weight of the Sr/ γ -alumina and Pt/ γ -alumina powders were mixed together and ball milled. Water was then added to the resulting material to produce a slurry. The slurry was applied to a cordierite monolith (400 cells/in²) to obtain a 25 wt. % loading on the monolith. The monolith was subsequently dried at 120°C to remove the water and calcined at 500°C in air for 6 hours.

[0022] Fig. 2 compares the NO_x sorption efficiency for the two platinum-strontium NO_x traps A and B made above. Trap A comprises the platinum and strontium loaded together on the same alumina while trap B comprises two separate phases in an intimate mixture: platinum on alumina and the strontium on other alumina. Both of these traps have identical amounts of platinum and strontium loadings on the monolith. From the figure, it can be seen that trap B (made according to an embodiment of the present invention) has better efficiency than trap A (not according to the present invention). It is believed that by providing the catalysts on separate support materials as in the present invention trap B, deleterious interactions between the platinum and alkaline metal are avoided which occur when they are provided together on the same alumina as in trap A.

Example 2

[0023] This example prepares another embodiment of a trap according to the present invention. Trap C of this example contains the two phases of platinum/ γ -alumina and platinum/ γ -alumina as two layers on the monolith. The nitrogen oxide trap efficiency of "layered" trap C is compared in Fig. 3 with that of trap B which uses similar catalysts but in an intimate mixture. Trap C was prepared as follows. 4 wt. % Pt/ γ -alumina and 20 wt. % Sr/ γ -alumina powders were prepared following the procedure for trap B. The Sr/ γ -alumina powder was ball milled and mixed with distilled water to produce a slurry of the material. The slurry was applied to a cordierite

monolith (400 cell/in²) to obtain a 12.5 wt. % loading of the material onto the monolith. The monolith was then dried at 120°C and calcined in air at 500°C. Next, the Pt/ γ -alumina powder was ball milled and mixed with distilled water to produce a slurry. This slurry was applied directly to the monolith carrying the Sr/ γ -alumina material and then dried and calcined. The finished loading of Pt/ γ -alumina on the monolith was roughly 12.5 wt. As seen in Fig. 3, the nitrogen oxide trapping ability of trap B which contained an intimate mixture of the catalyst phases was superior to that of trap C which contained distinct layers of the catalyst phases.

[0024] The data of nitrogen oxide trapping efficiency shown in Figs 2 and 3 was obtained by subjecting the traps to simulated lean burn exhaust gas containing: 600 ppm NO, 10% CO₂, 10% H₂O, 0.1% CO, 0.03% H₂, 50ppm C₃H₈, and 6% O₂. The total gas flow rate was 3 litres/min and the space velocity was 20,000hr⁻¹. NO_x trap sorption efficiency was averaged over a 5 minute lean sorption cycle.

Claims

1. A nitrogen oxide trap useful for trapping nitrogen oxide present in the exhaust gases generated during lean-burn operation of an internal combustion engine, said trap comprising distinct catalyst phases:
 - (a) a porous support loaded with catalyst comprising 0.1 to 5 weight % platinum; and
 - (b) another porous support loaded with 2 to 30 weight % catalyst of at least one material selected from the group consisting of alkali metal elements and alkaline earth elements, the weight % of catalyst being based on the weight of its porous support, and wherein the catalyst phases (a) and (b) are provided as an intimate mixture thereof.
2. A trap according to claim 1, wherein said porous support of (a) and said another porous support of (b) comprises γ -alumina.
3. A trap according to either claim 1 or claim 2, wherein said catalyst phase (a) further comprises components selected from ceria and rhodium.
4. A trap according to any one of the preceding claims, wherein the catalyst phase (a) comprises said first support loaded with 0.5 to 2 wt. percent platinum and the catalyst phase (b) comprises said another support loaded with 10 to 20 wt. percent said alkaline metal, each based on the weight of the respective porous support.
5. An internal combustion engine exhaust gas catalyst system comprising a nitrogen oxide trap as claimed

in any one of the preceding claims, the nitrogen oxide trap being arranged in the exhaust system and absorbing nitrogen oxides when the air/fuel ratio of exhaust gas flowing into said trap is lean, said nitrogen oxide trap releasing absorbed nitrogen oxides when the oxygen concentration in said exhaust gas is lowered.

6. An exhaust gas catalyst system according to claim 6, which further comprises a three way catalyst positioned upstream of the NO_x trap.
7. An exhaust gas catalyst system according to claim 5 or 6, which further comprises a three-way catalyst positioned downstream of the NO_x trap.
8. A process for trapping nitrogen oxides from the exhaust gases generated during lean-burn operation of an internal combustion engine, said process comprising the steps of: bringing said oxygen-rich exhaust gases, whose oxygen content is at the stoichiometric or more required for oxidising the components to be oxidised therein, into contact with a nitrogen oxide trap as claimed in any one of claims 1 to 4.
9. A process according to claim 9 which further comprises the step of contacting the exhaust gases with a three-way catalyst positioned upstream and/or downstream of the nitrogen oxide trap.

Patentansprüche

1. Eine zum Abfangen von in den -während des Magerverbrennungs-Betriebs eines Verbrennungsmotors erzeugten- Abgasen enthaltenen Stickoxiden nützliche Stickoxid-Falle, wobei diese Falle verschiedene Katalysatorphasen umfaßt:
 - (a) einen mit 0,1 bis 5 Gewichtsprozent Platin enthaltenden Katalysator beladenen, porösen Träger;
 - (b) einen anderen porösen Träger, der mit 2 bis 30 Gewichtsprozent eines Katalysators aus mindestens einem Material beladen ist, das aus der aus Alkalimetall-Elementen und Erdalkalimetall-Elementen bestehenden Gruppe ausgewählt ist; wobei die Gewichtsprozent des Katalysators auf dem Gewicht seines porösen Trägers basieren, und worin die Katalysatorphasen (a) und (b) als innige Mischung hiervon bereitgestellt werden.
2. Eine Falle nach Anspruch 1, in der der besagte poröse Träger von (a) und der besagte poröse Träger von (b) γ -Aluminiumoxid beinhalten.

3. Eine Falle nach entweder Anspruch 1 oder Anspruch 2, in der die besagte Katalysatorphase (a) weiterhin aus Cerdioxid und Rhodium ausgewählte Komponenten enthält. 5
4. Eine Falle nach einem der vorstehenden Ansprüche, in der die Katalysatorphase (a) den besagten ersten, mit 0,5 bis 2 Gewichtsprozent Platin beladenen Träger umfaßt; und in der die Katalysatorphase (b) den besagten anderen, mit 10 bis 20 Gewichtsprozent des besagten alkalischen Metalls beladenen Träger umfaßt; jeweils basierend auf dem Gewicht der entsprechenden, porösen Träger. 10
5. Ein Verbrennungsmotor-Abgaskatalysatorsystem, das eine Stickoxidfalle nach einem der vorstehenden Ansprüche umfaßt; wobei die Stickoxidfalle in dem Abgassystem angeordnet ist und Stickoxide absorbiert, wenn das Luft/Kraftstoff-Verhältnis des in diese Falle hineinströmenden Abgases mager ist; und wobei die besagte Stickoxid-Falle absorbierte Stickoxide freigibt, wenn die Sauerstoffkonzentration in diesem Abgas erniedrigt wird. 15
6. Ein Abgaskatalysator-System nach Anspruch 5, welches weiterhin einen oberstromig der NO_x -Falle angeordneten Drei-Wege-Katalysator umfaßt. 20
7. Ein Abgaskatalysator-System nach Anspruch 5 oder 6, welches weiterhin einen strömabwärts der NO_x -Falle angeordneten Drei-Wege-Katalysator umfaßt. 25
8. Ein Verfahren zum Abfangen von Stickoxiden aus während des Magerverbrennungs-Betriebs eines Verbrennungsmotors erzeugten Abgasen, wobei dieses Verfahren die Schritte umfaßt:
In Kontakt bringen dieser sauerstoffreichen Abgase, deren Sauerstoffgehalt stöchiometrisch oder größer ist als jener, der zur Oxidation der zu oxidierenden Komponenten darin erforderlich ist, mit einer Stickoxid-Falle nach einem der Ansprüche 1 bis 4. 30
9. Ein Verfahren nach Anspruch 8, welches weiterhin den Schritt umfaßt das Abgas mit einem Drei-Wege-Katalysator in Kontakt zu bringen, der oberstromig und/oder strömabwärts dieser Stickoxid-Falle angeordnet ist. 35
- (a) ein support poreux chargé de catalyseur comprenant de 0,1 à 5% en poids de platine; et
(b) un autre support poreux chargé à raison d'au moins 2 à 30% en poids de catalyseur à base d'au moins un matériau choisi dans le groupe constitué d'éléments métalliques alcalins et d'éléments alcalino-terreux, le poids en % du catalyseur étant calculé par rapport au poids du support poreux correspondant, et dans lequel les phases catalytiques (a) et (b) sont préparées sous forme d'un mélange homogène.
2. Piège selon la revendication 1, dans lequel ledit support poreux défini en (a) et ledit autre support poreux défini en (b) comprennent de la γ -alumine.
3. Piège selon la revendication 1 ou la revendication 2, dans lequel ladite phase catalytique (a) comprend en outre des composants choisis parmi l'oxyde de cérium et le rhodium.
4. Piège selon l'une quelconque des revendications précédentes, dans lequel la phase catalytique (a) comprend ledit premier support chargé à raison de 0,5 à 2% en poids de platine et la phase catalytique (b) comprend ledit autre support chargé à raison de 10 à 20% en poids dudit métal alcalin, la proportion de chaque élément étant calculée par rapport au poids du support poreux respectif.
5. Système catalytique de gaz d'échappement d'un moteur à combustion interne comprenant un piège à oxyde d'azote selon l'une quelconque des revendications précédentes, le piège à oxyde d'azote étant disposé dans le système d'échappement et absorbant des oxydes d'azote lorsque le rapport air/carburant du gaz d'échappement passant dans ledit piège correspond à un régime pauvre, ledit piège à oxyde d'azote libérant les oxydes d'azote absorbés lorsque la teneur dudit gaz d'échappement en oxygène est abaissée.
6. Système catalytique de gaz d'échappement selon la revendication 5, qui comprend en outre un catalyseur à trois voies installé en amont du piège à NO_x .
7. Système catalytique de gaz d'échappement selon la revendication 5 ou la revendication 6, qui comprend en outre un catalyseur à trois voies installé en aval du piège à NO_x .
8. Procédé pour piéger les oxydes d'azote dans les gaz d'échappement produits pendant le fonctionnement en régime pauvre d'un moteur à combustion interne, ledit procédé comprenant les étapes de :
mise en contact desdits gaz d'échappement

Revendications

1. Piège à oxyde d'azote utile pour piéger l'oxyde d'azote présent dans les gaz d'échappement pendant le fonctionnement en régime pauvre d'un moteur à combustion interne, ledit piège comprenant des phases catalytiques distinctes : 55

riches en oxygène, dont la teneur en oxygène est supérieure ou égale au niveau stoechiométrique requis pour oxyder les composants devant être oxydés dans ce gaz, avec un piège à oxyde d'azote selon l'une quelconque des revendications 1 à 4.

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9. Procédé selon la revendication 8, qui comprend en outre l'étape de mise en contact des gaz d'échappement avec un catalyseur à trois voies installé en amont et/ou en aval du piège à oxyde d'azote.

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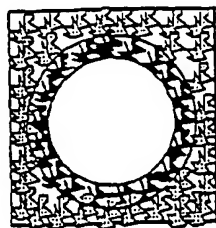


FIG.1A

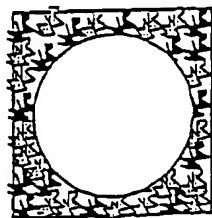




FIG.1B

 PLATINUM PHASE
 ALKALINE MATERIAL PHASE

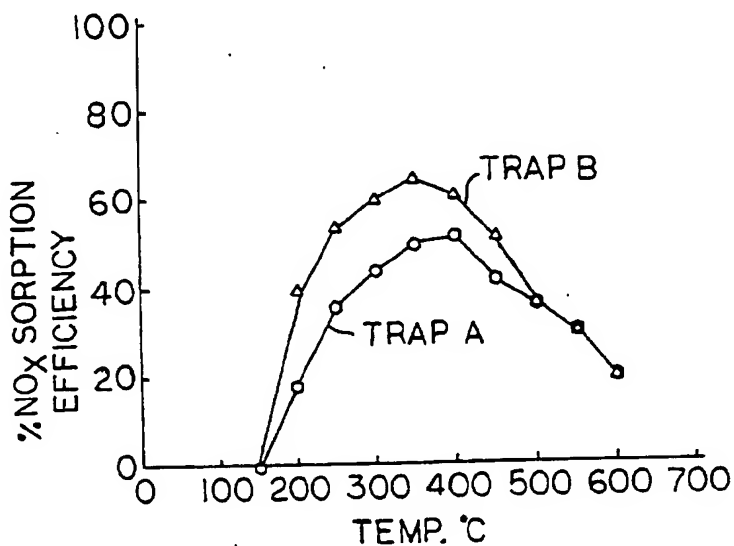


FIG.2

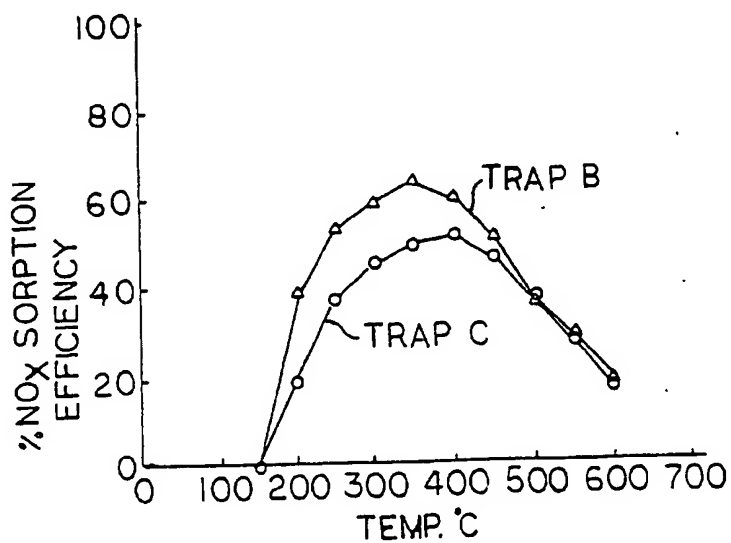


FIG.3